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PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Improved Process of Producing Anhydrous Substances

We, AUTOXYGEN, INC., a corporation organized under the laws of the State of New York, one of the United States of America, of 41, East 57th Street, New York City, New York, United States of America (Assignee of VAMAN RAMACHANDRA KOKATNUR, Chemist, of Rego Park, New York, United States of America, a citizen of the United States of America) do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

The production of anhydrous material from a solution of caustic soda, caustic potash or the like has heretofore presented numerous difficulties, has been an expensive process and has in most instances resulted in allegedly anhydrous end products which were difficult and hazardous to handle.

Several factors have rendered prior processes of drying such solutions or evaporating such solutions highly expensive. These include the use of elaborate equipment such as fusion pots, multiple effect evaporators and single effect evaporators. Fuel costs are high due to the temperatures at which the solutions have, of necessity, been treated by prior processes.

The removal of water from other materials such for instance as zinc chloride, magnesium chloride, magnesium sulphate, etc. wherein the water is held chemically rather than mechanically, presents still different problems and much expensive special apparatus has been designed for effecting the drying of such hydrolizable solutions. The formation of anhydrous end products from such solutions has heretofore involved still other difficulties due to the liberation of free acid as the solution hydrolizes at the elevated temperatures required for drying, such acid rapidly corroding the equipment.

We have discovered that in all such materials where drying has heretofore required excessively high temperatures (with or without hydrolysis and its undesirable consequences) can be dried most economically in simple standardized apparatus by using as an incident of such drying, a principle of partial pressure distillation.

According to this present invention we provide a method of producing anhydrous substances from deliquescent salts or hydroxides or salts capable of crystallizing with water of crystallization, wherein an aqueous solution of the salt is distilled in the presence of an inert diluent immiscible with water.

Kerosene is well suited for the inert diluent but equivalent hydrocarbons preferably having a boiling temperature of between 150° C. and 300° C. may be used. The solution and the added immiscible hydrocarbon diluent are distilled in an ordinary still until by partial pressure distillation all of the water which is mechanically or chemically combined with the desired end product, has been driven off.

The amount of diluent required to be distilled off to remove all of the water will vary with the different solutions to be dried and with the diluent used. However the amount of diluent which must be distilled off in any given case may be conveniently calculated by the following formula:—

$$\frac{mA}{m^1B} = \frac{PAMA}{PBM^1B}$$

In the foregoing formula A and B represent the two immiscible diluents, namely water and hydrocarbon; m and m^1 represent the weights of the two materials distilled off and M and M^1 represent the molecular weights of A and B and P represents the partial pressure of A and B at the temperature of distillation.

This process permits the removal of water at a temperature much lower than that which has previously been required to effect complete water extraction. One advantage of this is that where the materials under treatment are hydrolyzable at high temperatures, hydrolysis is reduced to a minimum. Consequently

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the corrosion of equipment by free acids which might be liberated is very substantially minimized. A contributory factor is not only the low temperatures employed but the protective action of the hydrocarbon diluent on the metal parts.

We have found that in using steel or iron equipment, corrosion in the process according to this invention is in the order of only $^1/_{10}$ to $^1/_{20}$ of the corrosion which occurs in prior processes utilizing higher temperatures.

The advantage of using standard iron equipment will of course be obvious. The use of the process effects a saving of about 50% of the manufacturing cost where caustic solutions are being dehydrated.

We shall now describe certain specific detailed examples of the manner in which our process may be economically carried out in practice. It is specifically to be understood however that these examples are given purely for illustrative purposes, the many variations of the process of these specific examples being wholly within the spirit and scope of the invention.

EXAMPLE I.

PRODUCING ANHYDROUS CAUSTIC SODA.

180 parts of a solution of caustic soda containing about 10% of caustic soda by weight, are mixed with 300 parts of kerosene and this mixture is distilled in a standard still. The mixture is agitated during gradual heating of the still and the water begins to distil off below 100° C., water removal being completed by the time the distillation temperature has approximated 200° to 225° C. In removing this water, about 126 parts of kerosene will have been distilled off, leaving 174 parts remaining in the still. The ten parts of caustic soda which remain behind in the still, together with the kerosene, are absolutely anhydrous and in granular form. Removal of the residue of the kerosene diluent may be effected by decanting or any other convenient or conventional manner. The granular caustic soda which remains contains less than a fraction of one per cent. of kerosene superficially adhering to the material but the presence of this negligible amount of kerosene has very definite advantage in the use of the final product. The slight kerosene residue adhering to the caustic soda particles aids in retarding corrosion of metallic containers in which the caustic soda may be packaged, renders the soda more stable and less corrosive to the skin and otherwise more generally suitable for commercial and household use.

Another advantage of our process, particularly as applied to production of anhydrous caustic soda, is the fact that the air in the manufacturing plant is not contaminated with caustic soda. There is less danger of skin irritation of the operatives in the plant. Caustic plants are notorious for accidents to employees not only in handling the caustic soda but during the standard process of fusing. The caustic soda produced by the present process is so stable that it can actually be carried in the hand for a short length of time without producing a burn and as above noted it does not get in the air, minimizes accidents in the household and prolongs the life of the tins in which it is packaged.

The amount of kerosene required to dehydrate the solution completely from solutions of various strengths, are roughly as follows:—

To remove 100 parts of water from a 4.77% caustic soda solution requires about 70 parts of kerosene. A 9% solution requires about 70 parts. A 14—25% caustic soda solution requires about 100 parts. More specifically

To make anhydrous NaOH:

%NaOH	Kerosene required to make 1 lb. NaOH	Lbs. Kerosene to remove 1 lb. H20
4.77%	14.2	0.71
9.1%	7.03	0.703
10.0	5.99	0.662
15	5.22	0.907
20	4.42	1.10

What is true with caustic soda solution is substantially true with caustic potash solutions, although the amounts of kerosene required for solutions of different strength necessarily vary.

EXAMPLE II.

PRODUCTION OF ANHYDROUS ZINC CHLORIDE.

500 parts of zinc chloride solution containing 600 parts of zinc chloride may be mixed with 600 parts of kerosene and the mixture heated and agitated in the still. After complete elimination of water, approximately 450 parts of kerosene will have been driven off, the water elimination occurring in the temperature range of from 212 to 360° F. The resultant product is white and granular and the hydrolysis of the chloride less than 1%.

EXAMPLE III.

PRODUCTION OF ANHYDROUS MAGNESIUM CHLORIDE.

240 parts of a solution containing 95 parts of magnesium chloride may be mixed with 500 parts of kerosene and

distilled in a standard still during the complete removal of water. Less than 380 parts of kerosene will be driven off as the still passes through the temperature range of 190 to 360° F. The hydrolysis of this process is about 13 to 14% of the hydrolysis which would normally occur with standard processes. The product is white and granular.

It should be borne in mind that while we have used kerosene as the hydrocarbon diluent in the illustrated examples, any equivalent material, having an approximately similar boiling range, may be substituted. The substitution of such materials such as naphthalene, dichlorbenzenes, dichlortoluenes or coal tar distillates and certain of their derivatives boiling at temperatures above 160° C., will necessarily involve adjustments in the amount of the diluent added to the solution to be dried, and in the amount of diluent driven off to effect complete removal of water and in the boiling range at which water is completely eliminated. All of the diluents, however, except certain chlorine derivatives seem to materially retard corrosion of the metal parts and the presence of a minute amount of diluent in the granular product in drying has beneficial effects.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A method of producing anhydrous substances from deliquescent salts or hydroxides or salts capable of crystallizing with water of crystallization, wherein an aqueous solution of the substance is distilled in the presence of an inert diluent immiscible with water.

2. A method according to claim 1, wherein the boiling point of the inert diluent lies between 150° C. and 300° C.

3. A method according to claim 1 or 2, wherein the diluent is a hydrocarbon or a hydrocarbon derivative.

4. A method according to claim 1, 2 or 3, wherein the diluent is kerosene.

5. A method according to any of the preceding claims, wherein the water produces at least 10% partial pressure of that of the diluent.

6. An anhydrous product whenever prepared by the process of manufacture particularly described and ascertained.

7. The process of producing anhydrous materials substantially as herein described.

Dated this 30th day of September, 1936.

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